

STANDARD OPERATING PROCEDURES: Column Leach Test

Methods: Standard Test Method for Leaching Solid Material in a Column Apparatus. ASTM D4874-95.

1.0. Scope and Application

This test method is a standard laboratory procedure for generating aqueous leachate from materials using a column apparatus. It provides a leachate suitable for organic analysis of semivolatile and nonvolatile compounds as well as inorganic analyses.

The column apparatus is designed and constructed of materials chosen to enhance the leaching of low concentrations of semivolatile and nonvolatile organic constituents as well as to maximize the leaching of metallic species from the solid. Analysis of column effluent provides information on the leaching characteristics of material under the conditions used in the test.

This test method provides for the passage of an aqueous fluid through materials of known mass in a saturated up-flow mode. It is intended that the sample used in the procedure be physically, chemically, and biologically representative of the material. This test method does not produce results that can be used as the sole basis for (a.) engineering design of a disposal site or (b.) the characterization of wastes based on their leaching characteristics.

This test method has the following limitations:

- 1.) Maximum particle size is 10mm (0.4 in.). Particle size reduction is not recommended. Large-diameter material should be removed prior to packing the column to ensure adequate compaction.
- 2.) Test materials containing densely immiscible organic material may result in phase separation and lead to column plugging.
- 3.) This test method does not differentiate between dissolved constituents and sub-70- μ m particulates that pass through the pores of the end plates.
- 4.) This test method is not applicable to the leachability characterization of materials with regard to volatile compounds.
- 5.) This test method is not applicable to the characterization of materials that dissolve in water to a degree that significantly impacts the void volume in the column or the determination of the specific gravity of the material.

Application of this test method to materials with initial low permeability, or to those that lose permeability over the course of the test, may result in long testing periods.

The values stated in SI units are to be regarded as the standard. The values given in parentheses are in approximate inch-pound equivalents.

2.0. Definitions

- 2.1. Reagent Water – water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest.
- 2.2. Void Volume - the volume between the solid particles in a bed of granular material. Also called the interstitial volume.

3.0. Equipment.

3.1. Apparatus

- 3.1.1. COLUMNS. The column body is constructed of glass pipe, 300 mm (12 in.) in length, with an inside diameter of 100 mm (4 in.). The cylinder wall must be of sufficient thickness, approximately 6 mm (1/4 in.), to withstand the operating pressure.

End plates are constructed of stainless steel. They are attached by means of eight, 6 mm (1/4 in.) threaded rods or any other means which ensures a leakproof seal.

Gaskets, one at each end of the column, are constructed of chemically inert materials, and are as thin as possible while still providing a good seal. The gasket diameter should overlap the inside column diameter by 3 mm (1/8 in.) to prevent the gasket from being forced out while under pressure. Techniques other than gaskets for providing a seal between the column and end plates are allowed provided the technique used is specified in the report.

Flow distribution disks must be constructed of sintered stainless steel, with a nominal pore diameter of 70 μm . The disk thickness shall be 6 mm (1/4 in.), with a diameter equal to the inside diameter of the column, approximately 100 mm (4 in.). The disk shall have eight evenly spaced grooves; each of which is 3 mm (1/8 in.) wide, 3 mm deep and 50 mm (2 in.) long. These grooves shall be positioned in a ray originating from the center of the disk.

Tubing used in the apparatus shall be of inert materials such as glass or stainless steel. The outer diameter shall be 6 mm (1/4 in.).

Substitution of materials of construction of the column or any of its parts is acceptable as long as it is demonstrated that levels of contamination for analytes of interest are equal to or less than those specified. Any modification of the apparatus as described in this test method must be justified, documented, and delineated in the report.

3.1.2. **PRESSURIZED RESERVOIR VESSEL.** This is used to contain the leaching fluid, is constructed with requirements similar to those of the leaching column with the following exceptions: (a.) no diffusion disks are used, and (b.) it is equipped with a top port for refilling the fluid.

3.1.3. **BALANCE.** A 10-kg capacity, with a 1-g sensitivity.

3.1.4. **COMPRESSED GAS SOURCE.** A source of pre-purified nitrogen or argon with a two-stage delivery regulator (0 to 350 kPa) (0 to 50 psig), and a pressure gage capable of measuring the pressure in the head space of the liquid reservoir to within 7 kPa (1psig).

4.0. Documentation

5.0. In-house Modifications to Referenced Method

6.0. Procedures

6.1. Preconditioning:

6.1.1. Prepare a test portion of waste in a manner that simulates the state the waste is in or will be in as it undergoes leaching in the field. Preparation of the test portion may include such factors as curing, and adjustment of moisture content and density. For such adjustments, the following procedures can be used when appropriate

- 6.1.2. Moisture Content. Adjust the moisture content to that defined in the disposal scenario by dewatering or adding reagent water to the material. Increase moisture content to that defined in the disposal scenario by addition of reagent water. Note and record the volume of reagent water added. If the concentration of any trace analyte of concern found in the reagent water exceeds the reporting (quantitation) limit for the volume added, note this on the report form. Decrease moisture content by determining scenario temperature (normally less than 60 degrees C) and drying the material at the temperature for a specified time interval. Determine actual moisture content of the waste as it is placed in the column. Note and record the drying temperature used. Record the moisture content.
- 6.1.3. Density. Density is to be adjusted by vibration or compaction to the anticipated field density. Pack the waste material in the column so that uniform density is achieved. This can be checked by visual observation of the waste in the transparent column.
- 6.1.4. Curing. Aging may be necessary for those materials that undergo physical or chemical changes with time in order to obtain a specimen (test portion) representative of the desired conditions. Curing may be accomplished in the field before testing in the column to produce a physical and chemical state representative of the waste as it undergoes leaching in the field. Note and record field conditions of temperature, humidity, and atmospheric pressure. Record the duration of the curing process and report with the test data.
- 6.1.5. Particle Size. Ensure that the particle size distribution of the waste as placed in the column is representative of that expected in field placement. Interpretation of results is based in part on knowledge of the particle size distribution and surface area of the material. Maximum particle diameter must not exceed 1/10 of the inside diameter of the column. Particle size reduction is not recommended. An evaluation of the particle characteristics of size distribution and surface area may be useful.
- 6.2. Column Filling:
- 6.2.1. Fill the column by compacting or vibrating the waste in five approximately equal layers. Approximate the expected field density. Scarify the top surface of each layer before adding the next increment. Do not scarify the top layer after final compaction. Fill the column to its final height, ensuring that the final height of the compacted material is equal to the height of the leaching column.
- 6.2.2. After filling the column, determine the mass, including the same fittings as in the determination of the tare. Record the total mass.
- 6.3. Leaching:
- 6.3.1. The leaching process is conducted in a continuous up-flow mode.
- 6.3.2. Saturation. After assembling the column, saturate the column with reagent water by the method used for determining permeability coefficients using a constant head permeameter. In this case, the column assembly replaces the constant head permeameter of that procedure. If it is determined that it is not possible to saturate the waste, it may be necessary to increase the vacuum used in the procedure or attempt to saturate the column under pressure, or both, to promote the dissolution of gasses.
- 6.3.3. Water used to saturate the waste should be of the same quality as that to be used to leach the waste in the column. Collect a sample of this water at the time of

saturation and analyze for the same properties and constituents as will be measured in the column effluent. An appropriate sample container, similar to that used for sample collection should be chosen and cleaned. Sample container guidance is given in 6.6.1. The test portion will be collected using the procedures for leachate collection.

- 6.3.4. Determination of the Void Volume. Void volume is estimated from the measurement of specific gravity of the solids, the mass of the wet solids placed into the column, the moisture content of the material, and the volume of the column. Although the drying temperatures used can be selected on the basis of the specific characteristics of the material being dried, the drying temperatures used in the procedures must be the same for the data to be used for porosity and void volume calculations.

- 6.3.4.1 Measure the mass of the sample material that fills the column apparatus. Note and record the moisture content of the material, as well as the specific gravity and any other specific information relevant to the material.

- 6.3.4.2 Calculate the void volume using the following equation:

$$V_p = V_c \{M / [(1+w) \times S \times D]\}$$

Where:

V_p = void volume in the column, cm^3

V_c = volume of the column, cm^3

M = as-packed mass of the material, including moisture, contained in the column

w = moisture content of the material contained in the column as a mass ratio g water/g solids

S = specific gravity of the material, unitless

D = density of water, g/cm^3

- 6.3.5. Estimate the degree of saturation by calculating the porosity n of the material using the following equation:

$$n = V_p / V_c$$

Calculate the mass gain in the saturated material. Add the value of the mass gain to the mass of the water in the packed column. Compare the value to the calculated void volume. The ratio is the degree of saturation. The degree of saturation can be estimated by determining the mass gain due to the water added to the waste during the saturation process, adding this mass gain to the mass of water in the packed column before saturation, and comparing this to the void volume just calculated. The mass gain can be determined by subtracting the mass of the packed, unsaturated column plus the mass of the packed, unsaturated column plus the mass of the water retained in the end plates, flow distribution disks, and fittings, from the mass of the saturated column.

6.4. Effluent Flow:

- 6.4.1. Once the column is saturated, begin the testing period. Adjust the pressure or hydraulic head, or both, so that one complete void volume exchange rate is accomplished in 24 hours (plus or minus 3 hours). Maximum operating column pressure, for apparatus specified, should not exceed 275 kPa (40 psig). Read and record operating pressure, and effluent start time. If some other test period is

warranted, record justification, time interval required, operating conditions, and effluent start time. Collect all column effluents as discrete void volume increments.

6.4.2. Operating Requirements. The column must be operated continuously. Note and record stop and restart times for all cessations. Determine total stoppage time for each occurrence per day and total daily stoppage. Cease test if, on any one day, total stoppage exceeds 1 h.

6.4.3. No type of channeling is permitted. When channeling is noted, note the time and cease the test. Determine if samples generated previously are usable. Document rationale for acceptance/rejection of previous sample effluent. Columns shall be protected from extraneous light except during column checks and sample collection intervals. Note and record each period of exposure.

6.4.4. Columns are to be run at ambient temperature, under normal test conditions. Sufficient pressure is required to maintain flow at the specified rate that will yield a void volume during the specified time interval. Temperature and pressures must be read and recorded periodically. For safety reasons, do not operate this equipment above 275 kPa (40 psig).

6.5. Leachate Collection:

6.5.1. The column must be inspected periodically and adjustment made if necessary to maintain the desired operating conditions.

6.5.2. Sufficient leachate from void volume periods shall be collected to establish a basis for judgment on completeness of the test. Minimally, it is recommended that Void Volumes 1,2,4, and 8 be collected and analyzed. Additional void volumes may provide additional useful data. The minimum number and sequence numbers of void volumes that will be collected and analyzed must be identified in advance of any collection activity.

6.5.3. Void volumes are sampled by collecting all column effluent generated over a 24-hour or other period corresponding to the void volume of interest.

6.5.4. Minimize contact of the leachate with the atmosphere. Fill effluent collection vessel headspace with pre-purified nitrogen or argon. Protect vessel headspace with a water trap.

6.5.5. Collect effluent in appropriate containers. Containers shall be cleaned in an appropriate manner. Containers must be free from contamination for those parameters of interest in the leachate.

6.6. Leachate Sample Preparation:

6.6.1. Sample containers must be constructed of materials that do not alter the sample quality. Sample containers must be cleaned as appropriate for the constituents of interest.

6.6.2. If collected leachates from void volume periods are stirred or agitated before filtration or analysis, this must be noted in the report. Minimize contact between the liquid and air during this mixing. If there are solids visible in the collected leachate before or after mixing, this must be noted in the report.

6.6.3. If filtration of the leachate is desired prior to chemical analyses, then such filtration steps must be noted. Analyze the filtered material separately to ensure that

the suspended solids did not contain constituents that are of environmental significance. If suspended solids are found to contain analytes of significance, then redo the column leach test, and analyze the leachate without filtration.

- 6.6.4. If samples are filtered before analysis, the method of filtration must be noted in the report. Filtration must be completed within 1 hour of sample collection. Contact with air must be minimized.
- 6.6.5. Samples must be stored under refrigeration at 4 degrees C. Sample container headspace must be minimized. A tight seal between the cap and the container is expected.
- 6.6.6. Sample Labeling. At a minimum, the information regarding column source, collection date, collection time, void volume number, sampler's name, sampler's signature, and preservatives used must be recorded.
- 6.6.7. Characterization of the Leachate. The minimum test portion for characterization is equal to the volume of leachate produced during the void volume period. Suggested analyses for characterization of leachate include any other analyses that will further characterize the material and provide interpretive guidance such as pH, Redox Potential, Conductivity, and Total Solids.

7.0. Review

- 7.1. Supervisor reviews Service Request, enters information into the appropriate database and assigns sample to the analyst.
- 7.2. Analyst verifies Service Request, reviews the SOP, obtains the appropriate benchsheet and proceeds with the analysis. A handwritten benchsheet is generated and the data are then entered into the computer for data reduction.
- 7.3. The final computer generated benchsheet is placed into the job folder along with a copy of the handwritten data. The handwritten benchsheet and a copy of the final benchsheet are placed into the method folder in chronological sequence.
- 7.4. The supervisor reviews the job folder for completeness of analysis (all requested parameters have been run) and sufficiency of Quality Control.
- 7.5. Completed analysis is given to the Data Section for entry of data into LIMs and final report preparation.

8.0. Method References

Standard Test Method for Leaching Solid Material in a Column Apparatus. ASTM 2001. Method Number: D 4874-95.

